

REMARKS

The Examiner in patent Application Serial No. 09/302,270 stated that restriction to one of the following inventions was required under 35 U.S.C. 121:

- I. Claims 1-6, drawn to a pre-glass agglomeration, classified in class 512, subclass 1.
- II. Claims 7-20, drawn to a process for making a pre-glass agglomeration, classified in class 501, subclass 11.

In a telephone conference with Dolph Torrence, on January 10, 2000, Mr. Torrence made a provisional election with traverse on behalf of Applicant to prosecute the invention of Group I, claims 1-6. Although Mr. Torrence, of the Litman Law Offices, did not have Power of Attorney on this date, Applicant nonetheless provisionally elected with traverse the invention of Group I, claims 1-6, drawn to an agglomeration of fused microspheres (as amended), without prejudice to claims 7-20, drawn to a process for making an agglomeration of fused microspheres (as amended). Applicant respectfully disagrees with the Examiner's suggestion that the product as claimed can be made by an entirely different method. Application Serial No. 09/302,270 has been allowed, but has not yet issued.

Applicant has now filed this divisional application and, by the Preliminary Amendment, elects to cancel claims 1-6 drawn to an agglomeration of fused microspheres (as amended), and to prosecute claims 7-13 and 15-19, drawn to a process for making an agglomeration of fused microspheres (as amended). Applicant also elects to cancel claims 14 and 20.

Applicant's changes to the Specification are requested in order to more clearly convey that the instant application is drawn to a method of manufacturing microspheres, and not to the microspheres which are produced, as the microspheres which are produced are the subject of Application No. 09/302,270.

Applicant has included paragraph numbers in the Specification, as requested by the Final Rule Of The Changes To Implement The Patent Business Goals.

Applicant has included a designation in paragraph 1 to indicate that the present application is a division of co-pending Application No. 09/302,270, filed on April 30, 1999 and titled "Artificial Rock Fragrance Delivery System," by Jim Mosbaugh. Application No. 09/302,270 has been allowed but has not yet issued.

Applicant has amended the Specification at paragraph 26 to more clearly disclose that the step of drying occurs with a spray dryer with a diaphragm pump operating between 25 and 200 psi. Support for this change in the Specification may be found in claim 12 of the Specification as originally filed. Applicant has further amended the Specification at paragraph 26 to more clearly disclose that the use of a centrifugal atomizer is an alternative to the use of a two-fluid nozzle. Finally, Applicant has amended paragraph 26 to more clearly disclose that the step of drying occurs at about 100 °C to about 300 °C. Support for this change in the Specification may be found in claim 13 of the Specification as originally filed.

Applicant has amended the Specification at paragraph 27 to correct a typographical error, as the appropriate range of flow rates for the air stream in the furnace is 25-200 SCFH. Support for this change in the Specification may be found in Claims 10-13 of the claims as originally filed in Application No. 09/302,270, wherein flow rates of 50 - 200 SCFH are claimed, and in the originally filed Specification, at page 12, line 24, wherein it is disclosed that air flow rates of 25-100 SCFM are used. It should be noted that the use of flow rate units in "SCFM" as disclosed at page 12, line 14 of the originally filed Specification was a typographical error; the air flow rate should have been reported in "SCFH", as was used in Claims 10, 11, 12, and 13. No new matter has been added.

Applicant has also changed the Specification such as to eliminate "Example 2", and all references to "Example 2", located in the originally filed Specification at page 13, line 20 - page 14, line 15. A similar modification was made during the prosecution of co-pending Application No. 09/302,270.

Claims 7-20 have also been amended to more specifically describe Applicant's invention as drawn to a process for making an agglomeration of fused microspheres.

Claim 9 has been amended to replace the modifier "boron" with "boric acid", and to replace the modifier "calcium" with "calcium nitrate". Support for these modifications is found at page 11, line 23 of the originally filed Specification.

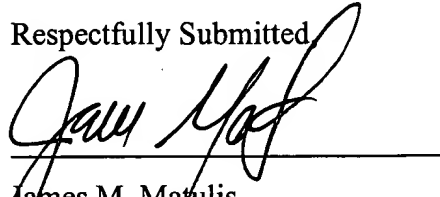
Claims 9-13 have also been amended to properly reflect that the appropriate air flow rates in the furnace are 25-200 SCFH, as discussed above. No new matter has been added.

Claims 14 and 20 have been canceled.

Lastly, the Title Sheet has been modified to reflect that Fowler, White has been substituted for the Litman Law Firm as the attorney representing the inventor for this Divisional Application.

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Respectfully Submitted



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"Version with markings to show changes made"

ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

CROSS-REFERENCE TO RELATED APPLICATION

[0001] The present application is a division of co-pending Application No. 09/302,270, filed on April 30, 1999 and titled "Artificial Rock Fragrance Delivery System," by Jim Mosbaugh. Application No. 09/302,270 has been allowed but has not yet issued. The present application claims the benefit of Application No. 09/302,270 and incorporates the contents by reference.

BACKGROUND OF THE INVENTION

1. FIELD OF THE INVENTION

[0002] The present invention relates to a method for making fragrance delivery systems [and a method for making the same]. In particular, the invention relates to a method of making pre-glass agglomerations that adsorb[s] fragrance producing oils and volatiles, and releases the fragrance innate to the oils and volatiles over an extended period of time without being messy or wet.

2. DESCRIPTION OF THE RELATED ART

[0003] Most delivery systems that utilize microspheres are manufactured out of acrylates or non-siliceous polymers. There are no fragrance delivery systems that utilize soda lime borosilicate microspheres fused together naturally without additives. Most fragrance systems have a short life span and lose their aroma within a few months. Virtually no currently available fragrance systems last for longer than a few months under any circumstances. Most also have a very intense smell initially with a reasonably pleasant odor after a few weeks which fades fairly fast.

[0004] Microspheres have been used in the past for a variety of purposes. The most common uses pertain to holders for chemicals in compositions such as holding fragrance for laundry detergent. In other words, the microspheres contain a chemical and are mixed with other compounds to form a heterogeneous composition where the microspheres will release the chemicals either gradually or all at once in response to a stimulus such as a change in ionic character, heat or other stimulus. Microspheres are also used in drug delivery systems designed to release the drug contained in the microsphere at a particular time according to pH or other factor.

[0005] The material and use of the pre-glass agglomeration [of]created by this invention are unique and unknown in the past. Also, these pre-glass agglomerations are not discrete spheres but rather modified soda-lime borosilicate sphere clusters, wherein thousands of microspheres become molecularly fused together via microcrystalline like structures on the sphere surfaces. Therefore, this invention provides a method of making a microsphere matrix without the addition of costly binders and polymers. These microcrystalline structures are distinctly different from current available['] industrially manufactured microspheres.

[0006] U.S. Patent No. 3,365,315 issued to Beck, et al. on January 23, 1968, discloses glass bubbles made from glass cullet particles by heating. This amorphous solid contains SiO_2 (60-80%), Na_2O (5-26%), CaO (5-25%), $\text{K}_2\text{O}/\text{Li}_2\text{O}$ (5-16%), and $\text{Na}_2\text{O}/\text{K}_2\text{O}/\text{Li}_2\text{O}$ (5-16%) plus some other oxides. The temperature range utilized for bubble formation is between 1050° and 1300°C. The resultant amorphous solid can be utilized as ingredients in molded parts designed for use in high pressure environments. These particles also have the capacity be used with thin walls thus possessing a maximum strength and crushable if that strength is exceeded. The methods utilized to make the glass bubbles taught by Beck, as well as the glass bubbles themselves, are very different from the rock of the present invention.

[0007] U.S. Patent No. 3,985,298 issued to Nichols on October 12, 1976, discusses controlled release materials, and method of using, that can be incorporated into a chemical delivery system. The materials utilized by Nichols are polymer-liquid composite materials which may contain 99% or

more of the liquid. These controlled release materials can be incorporated into aerosol propellants, food products, chewing gum, pharmaceutical compounds, agricultural products, or cosmetic preparations. The desired functions of the release materials are flavoring, scent, coloring, medication, is dermatological action, pesticidal action, or agricultural fertilizer. The materials and objectives utilized by Nichols are different from the present invention.

[0008] U.S. Patent No. 4,155,897 issued to Schlusener on May 22, 1979, discloses compositions exhibiting controlled release of an active substance. The compositions of Schlusener comprise an unsaturated polyester resin, an active substance, hollow microspheres of an organic material, and an inorganic material. The hollow microspheres can be made of glass and are mixed with an unsaturated polyester resin to make a molded solid or semisolid substance. An active ingredient, such as volatile oils, is added to the substance. The strength of the final product depends on the unsaturated polyesters used, but is less than the strength of the unsaturated polyester used because the hollow microspheres reduce the overall strength. The composition taught by Schlusener [is], and the method of making the composition, are different from the amorphous rock of the present invention, and the method of making it. The release of gas by the molded item is measured by a period of up to about half a year which is significantly less than the year and a half capacity of the present invention. There is a relatively high gas release rate the first week, less the next three weeks and even less for the remainder of the active time. Also, the compositions of Schlusener lacks the strength and low density combination of the present invention.

[0009] U.S. Patent No. 5,336,665 issued to Garner-Gray, et al. On August 9, 1994, discloses a hydrophobic porous inorganic carrier particle having a perfume absorbed into the particle. In particular, a detergent composition containing the carrier particle and a method for manufacturing the same is disclosed. The inorganic carriers used in Garner-Gray include aluminosilicates such as certain zeolites, clays, aluminas and silicas, all of which are chemically treated or naturally hydrophobic. These porous, inorganic carrier particles are not designed to release odor over an extended period of time, but to deliver perfume to clothing or other surface via a detergent or the like. The particles used in Garner-Gray are not designed for room deodorizers, are not strong, and are not exceptionally adsorbent in that they are hydrophobic and would not adsorb water or alcohols.

[0010] U.S. Patent No. 5,725,869 issued to Lo on March 10, 1998, describes microsphere reservoirs for controlled release applications. The microspheres, optionally containing an ingredient to be dispensed through controlled release, are prepared by solvent evaporation of an oil-in-water emulsion formed from an organic solvent containing a polymer and a plasticizer and an aqueous solution containing one or more emulsifying agents. The microcapsules formed are porous and spongy in structure as opposed to hollow. These microspheres have a relatively high load rate and a low dispersion rate. They are useful for agricultural chemicals, pharmaceuticals, cosmetics and fragrances. The invention of Lo is not designed to be a room deodorizer, and does not have a sturdy solid nature as does the rock of the current invention.

[0011] U.S. Patent No. 5,824,345 issued to Milstein on October 20, 1998, discloses compositions useful in the delivery of fragrances and flavorants. A method for preparing the compositions is disclosed: the active agent is mixed with the[,] proteinoid of hydrolyzed vegetable protein solution and the proteinoid or modified hydrolyzed vegetable protein is precipitated out of the solution, thereby forming a microsphere containing the active agent. The product formed by the method in Milstein differs from the present invention in that the present invention adsorbs any liquid, oil or alcohol, while Milstein requires the microspheres to be made concurrent with placing the agent therein which is a handicap in that it reduces the usefulness of the Milstein invention. Also, the microsphere of Milstein is not as sturdy as the current invention and[,] the aroma does not last nearly as long.

[0012] U.S. Patent No. 5,849,055 issued to Arai, et al. on December 15, 1998, discloses a process for making inorganic microspheres which comprises pulverizing a material by wet pulverization to obtain a slurry of a pulverized powder material, spraying the slurry to form liquid droplets, and heating the liquid droplets to fuse or sinter the powder material to obtain inorganic microspheres. These microspheres are discrete individual microbeads and cannot be utilized in the manner of the present invention. The microspheres of Arai can be used as a powder or an ingredient, but not as a deodorizing rock.

[0013] U.S. Patent No. 5,871,722 issued to Nacht, et al. on February 16, 1999, shows ionic beads useful for controlled release and adsorption. Active ingredients are released from the ionic polymer beads over an extended period of time such as when orally administered, or when applied to a keratinic material, typically human skin or hair, or when otherwise delivered to a target environment. Clearly, the ionic beads of Nacht are designed to deliver an active ingredient upon contact with some substance which releases their ionic bonds. These ionic beads would not be useful for room deodorants or absorption of oils.

[0014] U.S. Patent No. 5,534,348 issued to Miller, et al. on July 9, 1996, describes hollow borosilicate microspheres and a method of making them. The compositions of the sodium borosilicate starts with the preferred weight ratio of $\text{Na}_2\text{O}:\text{SiO}_2:\text{B}_2\text{O}_3$ between 1.0:2.5:0.2 and 1.0:3.22:0.5 for the starting material. The borosilicate microspheres of Miller are used in reflective paints and coatings, incorporated into molded plastic products, and for use as thermal insulation, but not as delivery vehicles for scents or as adsorbent materials.

[0015] None of the above inventions and patents, taken either singularly or in combination, is seen to describe the instant invention as claimed. Thus, a method of making an artificial rock fragrance delivery system solving the aforementioned problems is desired.

SUMMARY OF THE INVENTION

[0016] The current invention is a method for making a pre-glass agglomeration that acts as a vector for fragrance delivery by utilizing fused, microspheres with calcium integrated into the spheres from an aqueous sol precursor. The artificial fragrance delivery system is also referred to herein as an artificial rock fragrance delivery system, because the pre-glass agglomeration resembles a rock or rock-like structure. The fragrance delivery system has an extended fragrance release time generally exceeding a year and a half. The pre-glass agglomeration uses microcapillary action to quickly uptake oils and alcohols to more than double the weight of the pre-glass agglomeration. Also, the slow release of a fragrance without any residual liquid escape is another advantageous quality of the

instant pre-glass agglomeration invention. It is also possible to recharge or replenish the pre-glass agglomerations an unlimited number of times with additional fragrance oils/alcohols after the odor fades. The pre-glass agglomerations can also be molded into a variety of shapes using conventional vacuum applications to maintain the integrity of the resulting molded shape.

[0017] The pre-glass agglomerations with fragrance can be used for aromatherapy crock pots or boilers. They may also be dipped in flammable oils and used as incense or candle wicks. The pre-glass agglomerations can be used as room or facility fragrances, to[,] counteract pungent odors, and may be colored or dyed as desired.

[0018] The pre-glass agglomerations can also be used for various filtration applications. For example, they can be used for anionic and cationic separation by modification of the metal groups in order to bind salts from brine discharge in desalinization plants to economically reduce salt content. The pre-glass agglomerations can be used for separation filters for chemical processes such as removal of chlorine or sulfates from stacks. The surfaces, internal and external, of the pre-glass agglomerations can be modified by various acylations or substitutions to provide functional groups which can aid in separations, chemical collections and catalysis. Additional separation methods include the use of the rocks in saline/petroleum separation with for example oil spills.

[0019] There are numerous other uses as well. They can be used as low weight buoyancy control devices due to their extremely low density, and utilized as heat insulation and/or fire-resistant filler material. Also, they can be used as insect repellent by soaking the pre-glass agglomeration, having increased borate content, in citronella, lavender or other repellent. Sodium bicarbonate can be adsorbed by these rocks to produce fizzing in an essential oil bath. Naked or untreated pre-glass agglomerations can be used to adsorb various airborne chemical vapors, for example in nail salons and urethane production facilities.

[0020] Additionally, prior to high temperature processing, the powder may be applied to various fabrics in order to achieve water proofing characteristics. Thermal and sound insulation benefits also result. It is notable that in order to achieve flexibility from the coated fabric it is necessary to

incorporate various drying control chemical additives. The treated fabric is excellent for aeronautical applications such as the insulation barrier between the fuselage and the exterior of airplanes. Accordingly, it is a principal object of the invention to provide a method of making an agglomeration of pre-glass material that has exceptional absorption qualities, and is dry to the touch once dried and removed from the fluid to be adsorbed.

[0021] It is another object of the invention to provide a method of making an agglomeration of pre-glass material that can adsorb oils and other lipophilic substances readily without significant mess. It is a further object of the invention to provide a method of making an agglomeration of pre-glass material that can adsorb alcohol-based liquids readily. Still another object of the invention is to provide a method of making an agglomeration of pre-glass material that after absorption of an aromatic oil /alcohol-based substance will release the fragrance of the adsorbed substance over an extended period of time. It is an object of the invention to provide a method of making improved elements and arrangements thereof for the purposes described which is inexpensive, dependable and fully effective in accomplishing its intended purposes. These and other objects of the present invention will become readily apparent upon further review of the following specification.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0022] The present invention pertains to a highly adsorbent pre-glass agglomeration or rocks which can be utilized in diverse ways. The pre-glass agglomeration can be used to adsorb oil/alcohol-based liquids. Upon absorption of liquids, the surface of the pre-glass agglomeration is dry to the touch, thus eliminating any potential mess or stickiness. The pre-glass agglomeration is a long term fragrance delivery system that will adsorb aromatic oil/alcohol based liquids, and then release the fragrance of the aromatic liquid slowly over a sustained period of time, generally up to about one and a half years.

[0023] Colored liquid(s) and/or dye(s) can be used to decorate the pre-glass agglomeration, wherein the resulting pre-glass agglomeration has the color or dye of the[,] liquid adsorbed therein.

Also, the pre-glass agglomeration can be formed into various shapes as desired [o]. Other applications include salt binding, filtration, separation and insulation. As used herein, all percentages (%) are percent weight in volume of water prior to heating, also expressed as weight/volume %, %(w/v), w/v, w/v% or simply %, unless otherwise indicated. The following [examples are] example is the preferred embodiment[s] of the pre-glass agglomeration according to the present invention. It should be noted, however, that the[s] example[s] is [are] by no means a limitation[s] of the invention, and that various modifications and improvements in the manufacturing process all fall under the scope of this invention.

Example 1

SiO ₂	from about 60 to about 75%;
Na ₂ O	from about 10 to about 35%;
K ₂ O	from about 2 to about 20%;
B ₂ O ₃	from about 5 to about 20%; and
CaO	from about 0.5 to about 12%

[0024] Preferably, commercial silicates are utilized such as sodium silicate having a weight ratio 3.22, or sodium silicate modified with a caustic agent or acetate having a weight range between 2.8-3 silicate to alkali, or potassium silicates such as KASIL (PQ Corporation) having a weight ratio 2.44 are used. Modifiers such as tech grade boric acid and calcium nitrate are also used. The slurry for the modifiers is approximately 8-18% solids. The total solution is between 20-40% solids. Other modifiers may be added in quantities from about 1-10% These other modifiers may include Pb, MgO, Al₂O₃, BaO, Li₂O, Ge, and S.

[0025] A preferred method of making the pre-glass agglomeration of Example 1 comprises the following steps: The constituents are mixed together in two separate factions comprising the silicate part and the modifier part. The modifier part is boric and calcium in an aqueous slurry. The modifier solution is either poured into the silicate solution with vigorous mixing or the two are mixed together using an impeller pump with a recirculation loop. Vigorous mixing and slow addition of the boric/calcium solution are essential.

[0026] The solution, once mixed together, has a pH of 10-12. Mixing temperatures approach 60°C. This solution is fed to a two fluid nozzle for atomization via a diaphragm pump at 25-200 psi. [Also] Alternatively, a centrifugal atomizer [is] may be utilized at 10,000-25,000 rpm. While air atomizing, air pressure varies between 80-1000 psi. The drying step occurs at about 100 °C to about 300 °C. The outlet temperature is 300°-800°F.

[0027] The spray-dried product is then fed via pneumatic conveyor to a rotary tube furnace. The powder is fed into the furnace via an Accurate Feeder to a 316 SS tube rotating at 7-12 rpm and an angle of repose approximately 1/8 to 5 inches per foot. The furnace has 4 discrete zones with a temperature profile from 200°C to 1200°C with either a co-current or a counter current dry air flow at approximately [25 - 100 SCFM] 25 - 200 SCFH. Another atmosphere which is reducing, for example methane, may be used.

[0028] The pre-glass agglomeration is then collected from the furnace and sifted to remove any free flowing spheres from the pre-glass agglomeration. As a result of the sifting, the pre-glass agglomeration takes on a smooth surface. Examination under 40X microscopy indicated thousands of fused spheres.

[0029] The pre-glass agglomeration, once formed, is dipped into a solution containing various fragrance(s) or essential oils and allowed to soak for approximately 20-30 minutes. Conversely, the pre-glass agglomeration can be placed in a shallow dish of oils and inserted into a high pressure oven at ambient temperatures to reduce absorption time. The oils may also be dyed to impart color to the finished rock. The pre-glass agglomeration is removed from the dip via a screening process and conveyed under a series of ultraviolet heat lamps in order to dry the pre-glass agglomeration to the touch.

[0030] Oils used in the absorption process are preferably cut with a carrier such as dipropylene glycol, propylene glycol, SD alcohols, etc. Pre-glass agglomerations to be used in contact with the skin will use only FDA approved carriers and oils.

[Example 2]

SiO₂ from about 60 to about 75%;
Na₂O from about 10 to about 35%;
K₂O from about 2 to about 20%;
B₂O₃ from about 5 to about 20%;
CaO from about 0.5 to about 12%; and
HNO₃ from about 3 to about 25%

An alternative process for making the pre-glass agglomeration is as follows. Mix in the same amounts as used in Example 1 except a high speed mixer is used, with no impeller pump. To this solution, add HNO₃ with vigorous mixing until a gel is obtained. In addition to HNO₃, other acids may be utilized such as H₂SO₄, HCl, H₃PO₄, HF, and acetic acid.

The solution is then heated on a shelf oven at 100°-300°C to remove the water. The resultant frit is subsequently ball or screen milled to obtain particles approximately 20-100 microns in diameter. The particles are run through the rotary tube furnace as mentioned in Example 1, forming agglomerations where the individual particles are not necessarily hollow spheres but capillaries allow for adequate saturation of the oils and are held in place by weak bond forces with the same result. The pre-glass agglomeration is dipped in the same way as before to make an aromatic article.]

[0031] Pre-glass agglomerations made by [either of]the above method[s] can also be used to separate oil from saline. Pre-glass agglomerations which have not been soaked in a fragrance containing liquid are preferably used for this.

[0032] In order to separate oil from saline the pre-glass agglomerations are placed in an oil and water dispersion and are mixed either by tidal action or paddle. Allow the agglomerations to soak for up to 48 hours and then collect the agglomerations with a screen or net. Place the agglomerations in either a conventional oven or a vacuum oven in order to reclaim the petroleum. The reclaimed petroleum can then be recycled. The agglomerations can be discarded or reused. To reuse, the agglomerations must be washed with a low weight alcohol.

[0033] It is to be understood that the present invention is not limited to the embodiments described above, but encompasses any and all embodiments within the scope of the following claims.

CLAIMS

I claim:

- [1. A pre-glass agglomeration, comprising:
SiO₂ about 60 to about 75%;
Na₂O about 10 to about 35%;
K₂O about 2 to about 20%;
B₂O₃ about 5 to about 20%; and
CaO about 0.5 to about 12%.
2. The pre-glass agglomeration of claim 1, further comprising:
a liquid selected from the group consisting of an oil and an alcohol.
3. The pre-glass agglomeration of claim 2, wherein:
said liquid is a fragrance.
4. The pre-glass agglomeration of claim 1, further comprising:
about 20% of an acid selected from the group consisting of HNO₃, H₂SO₄, HCl, H₃PO, HF
and acetic acid.
5. The pre-glass agglomeration of claim 4, further comprising:
a liquid selected from the group consisting of an oil and an alcohol.
6. The pre-glass agglomeration of claim 5, wherein:
said liquid is a fragrance.]
7. (amended) A process for making [a pre-glass]an agglomeration[,] of fused microspheres
comprising the steps of:
 - a. mixing silicates;
 - b. mixing modifiers;

- c. mixing silicates and modifiers together to form a mixture;
d. drying the mixture to form a dry resultant material;
e. collecting the dry resultant material;
f. heating the resultant material to form [-a pre-glass]an agglomeration; and
g. collecting the [pre-glass] agglomeration.

8. (amended) The process for making [a pre-glass]an agglomeration of fused microspheres as in claim 7, further comprising the steps of:

- a. soaking the [pre-glass] agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
b. removing the [pre-glass] agglomeration from the liquid fragrance; and
c. drying the fragrance containing [pre-glass]the agglomeration wherein said drying is selected from the group consisting ultra violet light or heat.

9. (amended) A process for making [a pre-glass]an agglomeration of fused microspheres as in claim 7, wherein:

said silicates are sodium silicate and potassium silicate; and

said modifiers are [boron] boric acid, Pb, MgO, Al₂O₃, BaO, Li₂O, Ge, S and calcium nitrate.

10. (amended) A process for making [a pre-glass]an agglomeration of fused microspheres as in claim 9, wherein:

- a. the step of mixing the silicates and the modifiers together to form the mixture occurs by pouring the modifiers into the silicates;
b. the step of drying occurs with a spray dryer via a diaphragm pump at 50-150 psi and atomizing at 80 to 300 psi with outlet temperature ranging from about 300° to about 800°F; and
c. the step of heating the resultant material occurs in a furnace by an accurate feeder rotating 5-20 rpm at an angle of repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with a counter current dry air flow [50] 25 - 200 SCFH.

11. (amended) A process for making [a pre-glass]an agglomeration of fused microspheres as in claim 9, wherein:

a. the step of mixing the silicates and the modifiers together to form the mixture occurs by pouring the modifiers into the silicates;

b. the step of drying occurs with a spray dryer via a diaphragm pump at 50-150 psi and atomizing at 80 to 300 psi with outlet temperature ranging from about 300° to about 800°F; and

c. the step of heating the resultant material occurs in a furnace by an accurate feeder rotating 5-20 rpm at an angle of repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with a co-current dry air flow [50] 25 - 200 SCFH.

12. (amended) A process for making [a pre-glass]an agglomeration of fused microspheres as in claim 9, wherein:

a. the step of mixing the silicates and the modifiers occurs by an impeller pump and a recirculation loop;

b. the step of drying occurs with a spray dryer with a diaphragm pump at 25-200 psi and air atomizing at 80 to 800 psi with an outlet temperature ranging from about 300° to about 800°F; and

c. the step of heating the resultant material occurs in a furnace by an accurate feeder rotating 5-20 rpm at an angle of repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with a co-current dry air flow [50] 25 - 200 SCFH.

13. (amended) A process for making [a pre-glass]an agglomeration of fused microspheres as in claim 9, wherein:

a. the drying step occurs at about 100° to about 300°C; and

b. the step of heating the resultant material occurs in a furnace by an accurate feeder rotating 5-20 rpm at an angle of repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with a co-current dry air flow [50] 25 - 200 SCFH.

[14. (amended) A process for making a pre-glass agglomeration as in claim 9, further comprising:

a. adding about 20% acid to the mixture of silicates and modifiers by high shear vigorous mixing and ball milling to form the dry material, wherein said acids are HNO₃, H₂SO₄, HCl, H₃PO₄, HF, and acetic acid.]

15. (amended) The process for making [a pre-glass]an agglomeration of fused microspheres as in claim 9, further comprising the steps of:

a. soaking the [pre-glass] agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
b. removing the [pre-glass] agglomeration from the liquid fragrance; and
c. drying the fragrance containing [pre-glass]the agglomeration of fused microspheres wherein said drying is selected from the group consisting ultra violet light or heat.

16. (amended) The process for making [a pre-glass]an agglomeration of fused microspheres as in claim 10, further comprising the steps of:

a. soaking the [pre-glass] agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
b. removing the [pre-glass] agglomeration from the liquid fragrance; and
c. drying the fragrance containing [pre-glass]the agglomeration.

17. (amended) The process for making [a pre-glass]an agglomeration as in claim 11, further comprising the steps of:

a. soaking the [pre-glass] agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
b. removing the [pre-glass] agglomeration from the liquid fragrance; and
c. drying the fragrance containing [pre-glass]the agglomeration.

18. (amended) The process for making [a pre-glass]an agglomeration of fused microspheres

as in claim 12, further comprising the steps of:

- a. soaking the [pre-glass] agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
- b. removing the [pre-glass] agglomeration from the liquid fragrance; and
- c. drying the fragrance containing [pre-glass]the agglomeration wherein said drying is selected from the group consisting ultra violet light or heat.

19. (amended) The process for making [a pre-glass]an agglomeration of fused microspheres as in claim 13, further comprising the steps of:

- a. soaking the [pre-glass] agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
- b. removing the [pre-glass] agglomeration from the liquid fragrance; and
- c. drying the fragrance containing [pre-glass]the agglomeration wherein said drying is selected from the group consisting ultra violet light or heat.

[20. The process for making a pre-glass agglomeration as in claim 14, further comprising the steps of:

- a. soaking the pre-glass agglomeration in a liquid fragrance selected from the group consisting of an oil and an alcohol;
- b. removing the pre-glass agglomeration from the liquid fragrance; and
- c. drying the fragrance containing pre-glass agglomeration wherein said drying is selected from the group consisting ultra violet light or heat.]

ABSTRACT OF THE DISCLOSURE

sub 74
[0034] —[pre-glass agglomeration] A method of making a fragrance delivery system comprising forming fused microspheres and incorporating a fragrance therein. The method relates to the mixing together of two separate factions comprising a silicate part and a modifier part, drying the mixture, heating the mixture to form an agglomeration, removing any free-flowing spheres from the agglomeration, soaking the agglomeration in fragrances or essential oils, and then drying the agglomeration. [incorporated therein. The preglass agglomeration has an extended fragrance release time exceeding a year and a half, and uses microcapillary action to quickly uptake oils and alcohols. The pre-glass agglomeration provides a slow release of fragrance without the escape of any residual liquid. The pre-glass agglomeration may be replenished, an unlimited number of times, with fragrance containing oils and alcohols after the odor fades. The pre-glass agglomerations can be molded and may be colored or dyed.]

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Transmitted herewith for filing is the utility patent
application of: JIM MOSBAUGH

for: ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

Enclosed Are:


1. Patent Application, 25 sheets
2. Combined Declaration and Power of Attorney, 2 sheets
3. Verified Statement Claiming Small Entity Status, 1 sheet
4. Associate Power of Attorney, 1 sheet
5. Information Disclosure Statement
6. Form PTO-1449, with copies of 9 references
7. Filing Fee in the Amount of \$380.00

The filing fee has been calculated as shown below:

BASIC FEE	SMALL ENTITY	\$380.00
TOTAL CLAIMS	(20 - 20 = 0) x 9	0.00
IND. CLAIMS	(2 - 3 = 0) x 39	<u>0.00</u>
TOTAL		<u>\$380.00</u>

Additional fees due, if any, may be charged to Deposit Account
No. 12-1662 of the undersigned.

Respectfully Submitted,


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JRW

Docket No. 17186.00

IN THE APPLICATION

OF

JIM MOSBAUGH

FOR AN

ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

BACKGROUND OF THE INVENTION

1. FIELD OF THE INVENTION

The present invention relates to fragrance delivery systems and a method for making the same. In particular, the invention relates to a pre-glass agglomeration that adsorbs fragrance producing oils and volatiles, and releases the fragrance innate to the oils and volatiles over an extended period of time without being messy or wet.

2. DESCRIPTION OF THE RELATED ART

Most delivery systems that utilize microspheres are manufactured out of acrylates or non-siliceous polymers. There are no fragrance delivery systems that utilize soda lime borosilicate microspheres fused together naturally without additives. Most fragrance systems have a short life span and lose their aroma within a few months. Virtually no currently available fragrance systems last for longer than a few months under any circumstances. Most also have a very intense smell initially with a reasonably pleasant odor after a few weeks which fades fairly fast.

Microspheres have been used in the past for a variety of purposes. The most common uses pertain to holders for chemicals in

compositions such as holding fragrance for laundry detergent. In other words, the microspheres contain a chemical and are mixed with other compounds to form a heterogeneous composition where the microspheres will release the chemicals either gradually or all at once in response to a stimulus such as a change in ionic character, heat or other stimulus. Microspheres are also used in drug delivery systems designed to release the drug contained in the microsphere at a particular time according to pH or other factor.

The material and use of the pre-glass agglomeration of this invention are unique and unknown in the past. Also, these pre-glass agglomerations are not discrete spheres but rather modified soda-lime borosilicate sphere clusters, wherein thousands of microspheres become molecularly fused together via microcrystalline like structures on the sphere surfaces. Therefore, this invention provides a microsphere matrix without the addition of costly binders and polymers. These microcrystalline structures are distinctly different from current available industrially manufactured microspheres.

U.S. Patent No. 3,365,315 issued to Beck et al. on January 23, 1968 discloses glass bubbles made from glass cullet particles by heating. This amorphous solid contains SiO_2 (60-80%), Na_2O (5-26%), CaO (5-25%), $\text{K}_2\text{O}/\text{Li}_2\text{O}$ (5-16%), and $\text{Na}_2\text{O}/\text{K}_2\text{O}/\text{Li}_2\text{O}$ (5-16%) plus some other oxides. The temperature range utilized for bubble formation is between 1050° and 1300°C. The resultant amorphous solid can be utilized as ingredients in molded parts designed for use in high pressure environments. These particles also have the capacity to

be used with thin walls thus possessing a maximum strength and crushable if that strength is exceeded. The methods utilized to make the glass bubbles taught by Beck, as well as the glass bubbles themselves, are very different from the rock of the present invention.

U.S. Patent No. 3,985,298 issued to Nichols on October 12, 1976 discusses controlled release materials, and method of using, that can be incorporated into a chemical delivery system. The materials utilized by Nichols are polymer-liquid composite materials which may contain 99% or more of the liquid. These controlled release materials can be incorporated into aerosol propellants, food products, chewing gum, pharmaceutical compounds, agricultural products or cosmetic preparations. The desired functions of the release materials are flavoring, scent, coloring, medication, dermatological action, pesticidal action, or agricultural fertilizer. The materials and objectives utilized by Nichols are different from the present invention.

U.S. Patent No. 4,155,897 issued to Schlusener on May 22, 1979 discloses compositions exhibiting controlled release of an active substance. The compositions of Schlusener comprise an unsaturated polyester resin, an active substance, hollow microspheres of an organic material, and an inorganic material. The hollow microspheres can be made of glass and are mixed with an unsaturated polyester resin to make a molded solid or semisolid substance. An active ingredient, such as volatile oils, is added to the substance. The strength of the final product depends on the

unsaturated polyesters used, but is less than the strength of the unsaturated polyester used because the hollow microspheres reduce the overall strength. The composition taught by Schlusener is different from the amorphous rock of the present invention. The release of gas by the molded item is measured by a period of up to about half a year which is significantly less than the year and a half capacity of the present invention. There is a relatively high gas release rate the first week, less the next three weeks and even less for the remainder of the active time. Also, the compositions of Schlusener lacks the strength and low density combination of the present invention.

U.S. Patent No. 5,336,665 issued to Garner-Gray et al. on August 9, 1994, discloses a hydrophobic porous inorganic carrier particle having a perfume absorbed into the particle. In particular, a detergent composition containing the carrier particle and a method for manufacturing the same is disclosed. The inorganic carriers used in Garner-Gray include aluminosilicates such as certain zeolites, clays, aluminas and silicas, all of which are chemically treated or naturally hydrophobic. These porous inorganic carrier particles are not designed to release odor over an extended period of time, but to deliver perfume to clothing or other surface via a detergent or the like. The particles used in Garner-Gray are not designed for room deodorizers, are not strong, and are not exceptionally adsorbent in that they are hydrophobic and would not adsorb water or alcohols.

U.S. Patent No. 5,725,869 issued to Lo on March 10, 1998, describes microsphere reservoirs for controlled release applications. The microspheres, optionally containing an ingredient to be dispensed through controlled release, are prepared by solvent evaporation of an oil-in-water emulsion formed from an organic solvent containing a polymer and a plasticizer and an aqueous solution containing one or more emulsifying agents. The microcapsules formed are porous and spongy in structure as opposed to hollow. These microspheres have a relatively high load rate and a low dispersion rate. They are useful for agricultural chemicals, pharmaceuticals, cosmetics and fragrances. The invention of Lo is not designed to be a room deodorizer, and does not have a sturdy solid nature as does the rock of the current invention.

U.S. Patent No. 5,824,345 issued to Milstein on October 20, 1998, discloses compositions useful in the delivery of fragrances and flavorants. A method for preparing the compositions is disclosed: the active agent is mixed with the proteinoid of hydrolyzed vegetable protein solution and the proteinoid or modified hydrolyzed vegetable protein is precipitated out of the solution, thereby forming a microsphere containing the active agent. Milstein differs from the present invention in that the present invention adsorbs any liquid, oil or alcohol, while Milstein requires the microspheres to be made concurrent with placing the agent therein which is a handicap in that it reduces the usefulness of the Milstein invention. Also, the microsphere of

Milstein is not as sturdy as the current invention and the aroma does not last nearly as long.

U.S. Patent No. 5,849,055 issued to Arai et al. on December 15, 1998, discloses a process for making inorganic microspheres which comprises pulverizing a material by wet pulverization to obtain a slurry of a pulverized powder material, spraying the slurry to form liquid droplets, and heating the liquid droplets to fuse or sinter the powder material to obtain inorganic microspheres. These microspheres are discrete individual microbeads and cannot be utilized in the manner of the present invention. The microspheres of Arai can be used as a powder or an ingredient, but not as a deodorizing rock.

U.S. Patent No. 5,871,722 issued to Nacht et al. on February 16, 1999, shows ionic beads useful for controlled release and adsorption. Active ingredients are released from the ionic polymer beads over an extended period of time such as when orally administered, or when applied to a keratinic material, typically human skin or hair, or when otherwise delivered to a target environment. Clearly, the ionic beads of Nacht are designed to deliver an active ingredient upon contact with some substance which releases their ionic bonds. These ionic beads would not be useful for room deodorants or absorption of oils.

U.S. Patent No. 5,534,348 issued to Miller et al. on July 9, 1996, describes hollow borosilicate microspheres and a method of making them. The compositions of the sodium borosilicate starts with the preferred weight ratio of $\text{Na}_2\text{O}:\text{SiO}_2:\text{B}_2\text{O}_3$ between 1.0:2.5:0.2

and 1.0:3.22:0.5 for the starting material. The borosilicate microspheres of Miller are used in reflective paints and coatings, incorporated into molded plastic products, and for use as thermal insulation, but not as delivery vehicles for scents or as adsorbent materials.

None of the above inventions and patents, taken either singularly or in combination, is seen to describe the instant invention as claimed. Thus, an artificial rock fragrance delivery system solving the aforementioned problems is desired.

SUMMARY OF THE INVENTION

The current invention is a pre-glass agglomeration that acts as a vector for fragrance delivery by utilizing fused microspheres with calcium integrated into the spheres from an aqueous sol precursor. The artificial fragrance delivery system is also referred to herein as an artificial rock fragrance delivery system, because the pre-glass agglomeration resembles a rock or rock-like structure. The fragrance delivery system has an extended fragrance release time generally exceeding a year and a half. The pre-glass agglomeration uses microcapillary action to quickly uptake oils and alcohols to more than double the weight of the pre-glass agglomeration. Also, the slow release of a fragrance without any residual liquid escape is another advantageous quality of the instant pre-glass agglomeration invention. It is also possible to recharge or replenish the pre-glass agglomerations an unlimited

number of times with additional fragrance oils/alcohols after the odor fades. The pre-glass agglomerations can also be molded into a variety of shapes using conventional vacuum applications to maintain the integrity of the resulting molded shape.

5 The pre-glass agglomerations with fragrance can be used for aroma-therapy crock pots or boilers. They may also be dipped in flammable oils and used as incense or candle wicks. The pre-glass agglomerations can be used as room or facility fragrances to counteract pungent odors, and may be colored or dyed as desired.

10 The pre-glass agglomerations can also be used for various filtration applications. For example, they can be used for anionic and cationic separation by modification of the metal groups in order to bind salts from brine discharge in desalinization plants to economically reduce salt content. The pre-glass agglomerations
15 can be used for separation filters for chemical processes such as removal of chlorine or sulfates from stacks. The surfaces, internal and external, of the pre-glass agglomerations can be modified by various acylations or substitutions to provide functional groups which can aid in separations, chemical
20 collections and catalysis. Additional separation methods include the use of the rocks in saline/petroleum separation with for example oil spills.

 There are numerous other uses as well. They can be used as low weight buoyancy control devices due to their extremely low density, and utilized as heat insulation and/or fire-resistant filler material. Also, they can be used as insect repellent by

soaking the pre-glass agglomeration, having increased borate content, in citronella, lavender or other repellant. Sodium bicarbonate can be adsorbed by these rocks to produce fizzing in an essential oil bath. Naked or untreated pre-glass agglomerations can be used to adsorb various airborne chemical vapors, for example in nail salons and urethane production facilities.

Additionally, prior to high temperature processing, the powder may be applied to various fabrics in order to achieve water proofing characteristics. Thermal and sound insulation benefits also result. It is notable that in order to achieve flexibility from the coated fabric it is necessary to incorporate various drying control chemical additives. The treated fabric is excellent for aeronautical applications such as the insulation barrier between the fuselage and the exterior of airplanes.

Accordingly, it is a principal object of the invention to provide an agglomeration of pre-glass material that has exceptional absorption qualities, and is dry to the touch once dried and removed from the fluid to be adsorbed.

It is another object of the invention to provide an agglomeration of pre-glass material that can adsorb oils and other lipophilic substances readily without significant mess.

It is a further object of the invention to provide an agglomeration of pre-glass material that can adsorb alcohol-based liquids readily.

Still another object of the invention is to provide an agglomeration of pre-glass material that after absorption of an

aromatic oil/alcohol-based substance will release the fragrance of the adsorbed substance over an extended period of time.

It is an object of the invention to provide improved elements and arrangements thereof for the purposes described which is inexpensive, dependable and fully effective in accomplishing its intended purposes.

These and other objects of the present invention will become readily apparent upon further review of the following specification.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The present invention pertains to a highly adsorbent pre-glass agglomeration or rocks which can be utilized in diverse ways. The pre-glass agglomeration can be used to adsorb oil/alcohol-based liquids. Upon absorption of liquids, the surface of the pre-glass agglomeration is dry to the touch, thus eliminating any potential mess or stickiness. The pre-glass agglomeration is a long term fragrance delivery system that will adsorb aromatic oil/alcohol-based liquids, and then release the fragrance of the aromatic liquid slowly over a sustained period of time, generally up to about one and a half years.

Colored liquid(s) and/or dye(s) can be used to decorate the pre-glass agglomeration, wherein the resulting pre-glass agglomeration has the color or dye of the liquid adsorbed therein. Also, the pre-glass agglomeration can be formed into various shapes

as desired. Other applications include salt binding, filtration, separation and insulation.

As used herein, all percentages (%) are percent weight in volume of water prior to heating, also expressed as weight/volume%,
5 %(w/v), w/v, w/v% or simply %, unless otherwise indicated.

The following examples are the preferred embodiments of the pre-glass agglomeration according to the present invention. It should be noted, however, that these examples are by no means limitations of the invention, and that various modifications and
10 improvements in the manufacturing process all fall under the scope of this invention.

Example 1

SiO ₂	from about 60 to about 75%;
Na ₂ O	from about 10 to about 35%;
15 K ₂ O	from about 2 to about 20%;
B ₂ O ₃	from about 5 to about 20%; and
CaO	from about 0.5 to about 12%

Preferably, commercial silicates are utilized such as sodium silicate having a weight ratio 3.22, or sodium silicate modified
20 with a caustic agent or acetate having a weight range between 2.8-3 silicate to alkali, or potassium silicates such as KASIL (PQ Corporation) having a weight ratio 2.44 are used. Modifiers such as tech grade boric acid and calcium nitrate are also used. The slurry for the modifiers is approximately 8-18% solids. The total

solution is between 20-40% solids. Other modifiers may be added in quantities from about 1-10%. These other modifiers may include Pb, MgO, Al₂O₃, BaO, Li₂O, Ge, and S.

5 A preferred method of making the pre-glass agglomeration of Example 1 comprises the following steps. The constituents are mixed together in two separate factions comprising the silicate part and the modifier part. The modifier part is boric and calcium in an aqueous slurry. The modifier solution is either poured into the silicate solution with vigorous mixing or the two are mixed
10 together using an impeller pump with a recirculation loop. Vigorous mixing and slow addition of the boric/calcium solution are essential.

The solution, once mixed together, has a pH of 10-12. Mixing temperatures approach 60°C. This solution is fed to a two fluid
15 nozzle for atomization. Also, a centrifugal atomizer is utilized at 10,000-25,000 rpm. While air atomizing, air pressure varies between 80-1000 psi. The outlet temperature is 300°-800°F.

The spray-dried product is then fed via pneumatic conveyor to a rotary tube furnace. The powder is fed into the furnace via an
20 Accurate Feeder to a 316 SS tube rotating at 7-12 rpm and an angle of repose approximately 1/8 to 5 inches per foot. The furnace has 4 discrete zones with a temperature profile from 200°C to 1200°C with either a co-current or a counter current dry air flow at approximately 25-100 SCFM. Another atmosphere which is reducing, for example methane, may be used.

The pre-glass agglomeration is then collected from the furnace and sifted to remove any free flowing spheres from the pre-glass agglomeration. As a result of the sifting, the pre-glass agglomeration takes on a smooth surface. Examination under 40X microscopy indicated thousands of fused spheres.

The pre-glass agglomeration, once formed, is dipped into a solution containing various fragrance(s) or essential oils and allowed to soak for approximately 20-30 minutes. Conversely, the pre-glass agglomeration can be placed in a shallow dish of oils and inserted into a high pressure oven at ambient temperatures to reduce absorption time. The oils may also be dyed to impart color to the finished rock. The pre-glass agglomeration is removed from the dip via a screening process and conveyed under a series of ultraviolet heat lamps in order to dry the pre-glass agglomeration to the touch.

Oils used in the absorption process are preferably cut with a carrier such as dipropylene glycol, propylene glycol, SD alcohols, etc. Pre-glass agglomerations to be used in contact with the skin will use only FDA approved carriers and oils.

Example 2

SiO_2	from about 60 to about 75%;
Na_2O	from about 10 to about 35%;
K_2O	from about 2 to about 20%;
B_2O_3	from about 5 to about 20%;
CaO	from about 0.5 to about 12%; and
HNO_3	from about 3 to about 25%

An alternative process for making the pre-glass agglomeration is as follows. Mix in the same amounts as used in Example 1 except a high speed mixer is used, with no impeller pump. To this solution, add HNO_3 with vigorous mixing until a gel is obtained. In addition to HNO_3 , other acids may be utilized such as H_2SO_4 , HCl , H_3PO_4 , HF , and acetic acid.

The solution is then heated on a shelf oven at $100^\circ\text{--}300^\circ\text{C}$ to remove the water. The resultant frit is subsequently ball or screen milled to obtain particles approximately 20-100 microns in diameter. The particles are run through the rotary tube furnace as mentioned in Example 1, forming agglomerations where the individual particles are not necessarily hollow spheres but capillaries allow for adequate saturation of the oils and are held in place by weak bond forces with the same result. The pre-glass agglomeration is dipped in the same way as before to make an aromatic article.

Pre-glass agglomerations made by either of the above methods can also be used to separate oil from saline. Pre-glass agglomerations which have not been soaked in a fragrance containing liquid are preferably used for this.

In order to separate oil from saline the pre-glass agglomerations are placed in an oil and water dispersion and are mixed either by tidal action or paddle. Allow the agglomerations to soak for up to 48 hours and then collect the agglomerations with a screen or net. Place the agglomerations in either a conventional oven or a vacuum oven in order to reclaim the petroleum. The reclaimed petroleum can then be recycled. The agglomerations can

be discarded or reused. To reuse, the agglomerations must be washed with a low weight alcohol.

5 It is to be understood that the present invention is not limited to the embodiments described above, but encompasses any and all embodiments within the scope of the following claims.

CLAIMS

I claim:

1 1. A pre-glass agglomeration, comprising:

2 SiO₂ about 60 to about 75%;

3 Na₂O about 10 to about 35%;

4 K₂O about 2 to about 20%;

5 B₂O₃ about 5 to about 20%; and

6 CaO about 0.5 to about 12%.

1 2. The pre-glass agglomeration of claim 1, further
2 comprising:

3 a liquid selected from the group consisting of an oil and an
4 alcohol.

1 3. The pre-glass agglomeration of claim 2, wherein:
2 said liquid is a fragrance.

1 4. The pre-glass agglomeration of claim 1, further comprising:
2 about 20% of an acid selected from the group consisting of
3 HNO₃, H₂SO₄, HCl, H₃PO₄, HF and acetic acid.

1 5. The pre-glass agglomeration of claim 4, further
2 comprising:

3 a liquid selected from the group consisting of an oil and an
4 alcohol.

1 6. The pre-glass agglomeration of claim 5, wherein:
2 said liquid is a fragrance.

7. A process for making a pre-glass agglomeration, comprising the steps of:

- a. mixing silicates;
- b. mixing modifiers;
- c. mixing silicates and modifiers together to form a mixture;
- d. drying the mixture to form a dry resultant material;
- e. collecting the dry resultant material;
- f. heating the resultant material to form a pre-glass agglomeration; and
- g. collecting the pre-glass agglomeration.

1 8. The process for making a pre-glass agglomeration as in
2 claim 7, further comprising the steps of:

- 3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;
- 5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and
- 7 c. drying the fragrance containing pre-glass agglomeration
8 wherein said drying is selected from the group consisting ultra
9 violet light or heat.

1 9. A process for making a pre-glass agglomeration as in claim
2 7, wherein:

3 said silicates are sodium silicate and potassium silicate; and
4 said modifiers are boron, Pb, MgO, Al₂O₃, BaO, Li₂O, Ge, S and
5 calcium.

1 10. A process for making a pre-glass agglomeration as in
2 claim 9, wherein:

3 a. the step of mixing the silicates and the modifiers
4 together to form the mixture occurs by pouring the modifiers into
5 the silicates;

6 b. the step of drying occurs with a spray dryer via a
7 diaphragm pump at 50-150 psi and atomizing at 80 to 300 psi with
8 outlet temperature ranging from about 300° to about 800°F; and

9 c. the step of heating the resultant material occurs in a
10 furnace by an accurate feeder rotating 5-20 rpm at an angle of
11 repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with
12 a counter current dry air flow 50 - 200 SCFM.

↓
SCFM

gm
5/24/99 →

1 11. A process for making a pre-glass agglomeration as in
2 claim 9, wherein:

3 a. the step of mixing the silicates and the modifiers
4 together to form the mixture occurs by pouring the modifiers into
5 the silicates;

6 b. the step of drying occurs with a spray dryer via a
7 diaphragm pump at 50-150 psi and atomizing at 80 to 300 psi with
8 outlet temperature ranging from about 300° to about 800°F; and

9 c. the step of heating the resultant material occurs in a
10 furnace by an accurate feeder rotating 5-20 rpm at an angle of
11 repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with
12 a co-current dry air flow 50 - 200 SCFM.
H

1 12. A process for making a pre-glass agglomeration as in
2 claim 9, wherein:

3 a. the step of mixing the silicates and the modifiers occurs
4 by an impeller pump and a recirculation loop;

5 b. the step of drying occurs with a spray dryer with a
6 diaphragm pump at 25-200 psi and air atomizing at 80 to 800 psi
7 with an outlet temperature ranging from about 300° to about 800°F;
8 and

9 c. the step of heating the resultant material occurs in a
10 furnace by an accurate feeder rotating 5-20 rpm at an angle of
11 repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with
12 a co-current dry air flow 50 - 200 SCFM.
H

1 13. A process for making a pre-glass agglomeration as in
2 claim 9, wherein:

- 3 a. the drying step occurs at about 100° to about 300°C; and
4 b. the step of heating the resultant material occurs in a
5 furnace by an accurate feeder rotating 5-20 rpm at an angle of
6 repose 1/8 - 5 inches per foot at about 200°C to about 1200°C with
7 a co-current dry air flow 50 - 200 SCFM.

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1 14. A process for making a pre-glass agglomeration as in
2 claim 9, further comprising:

- 3 a. adding about 20% acid to the mixture of silicates and
4 modifiers by high shear vigorous mixing and ball milling to form
5 the dry material, wherein said acids are HNO₃, H₂SO₄, HCl, H₃PO, HF,
6 and acetic acid.

1 15. The process for making a pre-glass agglomeration as in
2 claim 9, further comprising the steps of:

- 3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;
5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and
7 c. drying the fragrance containing pre-glass agglomeration
8 wherein said drying is selected from the group consisting ultra
9 violet light or heat.

1 16. The process for making a pre-glass agglomeration as in
2 claim 10, further comprising the steps of:

3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;

5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and

7 c. drying the fragrance containing pre-glass agglomeration.

1 17. The process for making a pre-glass agglomeration as in
2 claim 11, further comprising the steps of:

3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;

5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and

7 c. drying the fragrance containing pre-glass agglomeration.

1 18. The process for making a pre-glass agglomeration as in
2 claim 12, further comprising the steps of:

3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;

5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and

7 c. drying the fragrance containing pre-glass agglomeration
8 wherein said drying is selected from the group consisting ultra
9 violet light or heat.

1 19. The process for making a pre-glass agglomeration as in
2 claim 13, further comprising the steps of:

3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;

5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and

7 c. drying the fragrance containing pre-glass agglomeration
8 wherein said drying is selected from the group consisting ultra
9 violet light or heat.

1 20. The process for making a pre-glass agglomeration as in
2 claim 14, further comprising the steps of:

3 a. soaking the pre-glass agglomeration in a liquid fragrance
4 selected from the group consisting of an oil and an alcohol;

5 b. removing the pre-glass agglomeration from the liquid
6 fragrance; and

7 c. drying the fragrance containing pre-glass agglomeration
8 wherein said drying is selected from the group consisting ultra
9 violet light or heat.

ABSTRACT OF THE DISCLOSURE

A pre-glass agglomeration fragrance delivery system comprising fused microspheres and a fragrance incorporated therein. The pre-glass agglomeration has an extended fragrance release time exceeding a year and a half, and uses microcapillary action to quickly uptake oils and alcohols. The pre-glass agglomeration provides a slow release of fragrance without the escape of any residual liquid. The pre-glass agglomeration may be replenished, an unlimited number of times, with fragrance containing oils and alcohols after the odor fades. The pre-glass agglomerations can be molded and may be colored or dyed.

COMBINED DECLARATION AND POWER OF ATTORNEY

As the below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

the specification of which is attached hereto unless the following box is checked:

☐ was filed on _____ as United States Application Serial Number or PCT International Application Number _____ and was amended on _____ (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificates, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)

Priority Not Claimed

(Number) (Country) (Day/Month/Year Filed)

☐

(Number) (Country) (Day/Month/Year Filed)

☐

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below.

(Application Number) (Filing Date)

(Application Number) (Filing Date)

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

(Application Number)	(Filing Date)	(Status -- patented, pending, abandoned)
(Application Number)	(Filing Date)	(Status -- patented, pending, abandoned)

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

Richard C. Litman:	Registration No. 30,868
Direct all telephone calls to:	Richard C. Litman (703) 486-1000
Address all correspondence to:	Richard C. Litman LITMAN LAW OFFICES, LTD. P.O. Box 15035 Arlington, VA 22215

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full Name of Sole or First Inventor: JIM MOSBAUGH

Signature: _____

Date: 4-26

Country of Citizenship: U.S.A.

Residence: 14724 Pine Glen Circle,

Lute, FL 33549

Post Office Address: Same

VERIFIED STATEMENT (DECLARATION) CLAIMING SMALL ENTITY STATUS
(37 CFR 1.9(f) and 1.27(b))--INDEPENDENT INVENTOR

As the below named inventor, I hereby declare that I qualify as an independent inventor as defined in 37 CFR 1.9 (c) for purposes of paying reduced fees under section 41 (a) and (b) of Title 35, United States Code, to the Patent and Trademark Office with regard to the invention entitled below and in:

TITLE OF INVENTION

ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

 X the specification filed herewith.

I have not assigned, granted, conveyed, or licensed and am under no obligation under contract or law to assign, grant, convey or license, any rights in the invention to any person who could not be classified as an independent inventor under 37 CFR 1.9 (c) if that person had made the invention, or to any concern which would not qualify as a small business concern under 37 CFR 1.9 (d) or a nonprofit organization under 37 CFR 1.9 (e).

Each person, concern or organization to which I have assigned, granted, conveyed, or licensed or am under an obligation under contract or law to assign, grant, convey, or license any rights in the invention is listed below:

 X no such person, concern or organization

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small business entity is no longer appropriate. (37 CFR 1.28 (b)).

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

JIM MOSBAUGH
Name of First Inventor


Signature of First Inventor

Date 4-26

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF:

APPLICANT : JIM MOSBAUGH

SERIAL NO. : Unassigned

ART UNIT: Unassigned

FILED : Herewith

EXAMINER: Unassigned

FOR : ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, DC 20231

Sir:

ASSOCIATE POWER OF ATTORNEY AND APPOINTMENT OF AGENTS
37 C.F.R. 1.34(b)

Please recognize as Associate Attorneys in this case:

John Remon Wenzel	Reg. No. 24,768
Charles K. Friedman	Reg. No. 39,195
William F. Lang, IV	Reg. No. 41,928
Robert B. Lyons	Reg. No. 40,708
Paula L. Craig	Reg. No. 40,295
Stephen J. Sand	Reg. No. 34,716
Ourmazd S. Ojan	Reg. No. 38,065

Please recognize as Associate Agents in this case:

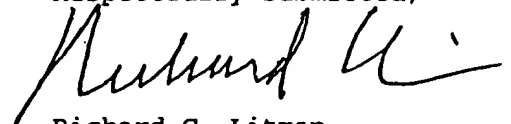
Dolph H. Torrence	Reg. No. 34,501
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Donald E. Watkins	Reg. No. 37,074
Warren S. Edmonds	Reg. No. 39,642
Edward G. Favors	Reg. No. 40,263
Thomas C. Schoeffler	Reg. No. 43,385

The addresses and phone numbers of the above Attorneys and Agents are the same as that of the undersigned Principal Attorney.

All previous Associate Powers are hereby revoked.

Please address all correspondence in this application to the undersigned Principal Attorney.

Respectfully submitted,



Richard C. Litman
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RCL:mjh

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN THE APPLICATION OF:

APPLICANT : JIM MOSBAUGH

SERIAL NO. : Unassigned

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EXAMINER: Unassigned

FOR : ARTIFICIAL ROCK FRAGRANCE DELIVERY SYSTEM

ASSISTANT COMMISSIONER OF PATENTS
WASHINGTON, DC 20231

Sir:

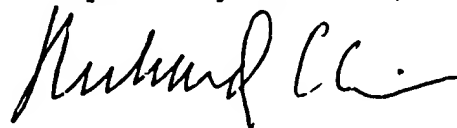
INFORMATION DISCLOSURE STATEMENT

35 U.S.C. 6; 37 C.F.R. 1.97; 1.98

Submitted herewith are copies of related art domestic patents, foreign patents and/or publications identified and discussed in the specification of the above identified application under the heading: "DESCRIPTION OF THE RELATED ART." These are identified on the attached PTO-1449 form.

The discussion of these related art patents and/or publications in the attached specification is believed to satisfy the duty to disclose requirements as set forth in the above identified statute and rules.

Respectfully submitted,



Richard C. Litman
Registration No. 30,868
(703) 486-1000

RCL:mjh

FORM PTO-1449 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE INFORMATION DISCLOSURE CITATION IN AN APPLICATION (Use several sheets if necessary)	ATTY. DOCKET NO. 17186.00	SERIAL NO.
	APPLICANT Jim MOSBAUGH	
	FILING DATE	GROUP

U.S. PATENT DOCUMENTS

Examiner Initials	DOCUMENT NUMBER	DATE	NAME	CLASS	SUBCLASS	FILING DATE if appropriate
	3,365,315	01/1968	Beck et al.			
	3,985,298	10/1976	Nichols			
	4,155,897	05/1979	Schlusener			
	5,336,665	08/1994	Garner-Gray et al.			
	5,534,348	07/1996	Miller et al.			
	5,725,869	03/1998	Lo			
	5,824,345	10/1998	Milstein			
	5,849,055	12/1998	Arai et al.			
	5,871,722	02/1999	Nacht et al.			

FOREIGN PATENT DOCUMENTS

DOCUMENT NUMBER	DATE	COUNTRY	CLASS	SUBCLASS	Translation YES NO

OTHER DOCUMENTS (Including Author, Title, Date, Pertinent Pages, etc.)

EXAMINER	DATE CONSIDERED
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EXAMINER: Initial if citation considered, whether or not citation is in conformance with MPEP 609; Draw line through citation if not in conformance and not considered. Include a copy of this form with next communication to applicant.